Thermochemical and microstructural contributions of high temperature particle erosion durability in CMAS exposed EBCs

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[3]University of Toledo

Acknowledgments
Transformational Tools and Technologies (TTT) Project
Hybrid Thermally Efficient Core (HyTEC) Project

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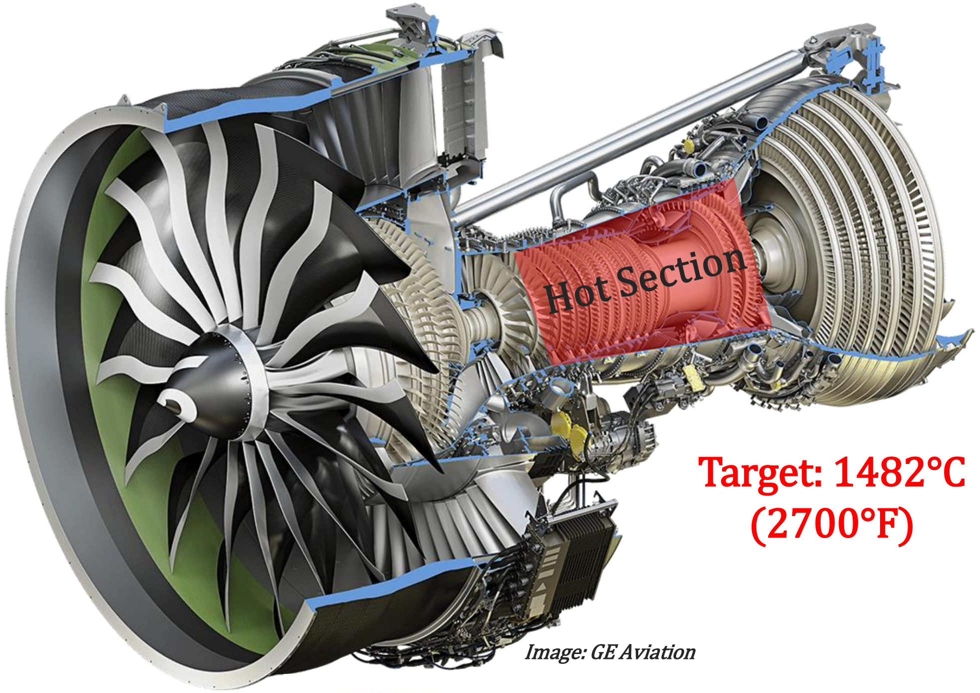
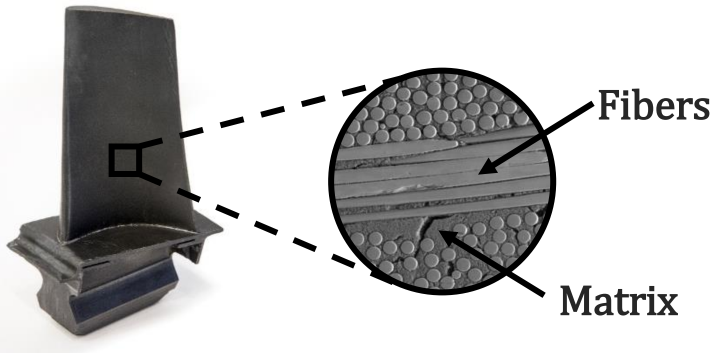
HILTON DAYTONA BEACH RESORT AND OCEAN CENTER
DAYTONA BEACH, FL, USA

Introduction and Motivation – Ceramic Matrix Composites (CMCs)

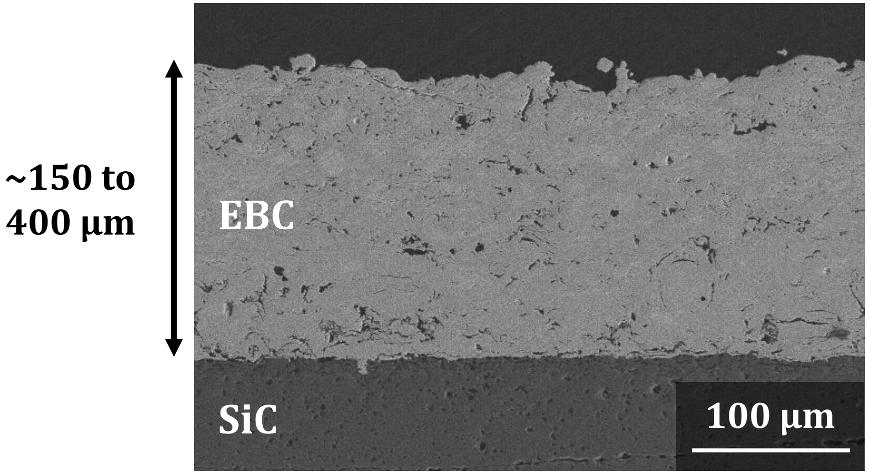


Target: 1482°C (2700°F)

CMC Turbine Blade



- Silicon carbide (SiC) CMCs susceptible to environmental attack at temperatures >800°C in oxygen and water vapor
    - Silica (SiO_2) scale formation that volatilizes in H_2O environment
    - Surface recession
- Require **environmental barrier coatings (EBCs)** to protect CMC component from harsh environment



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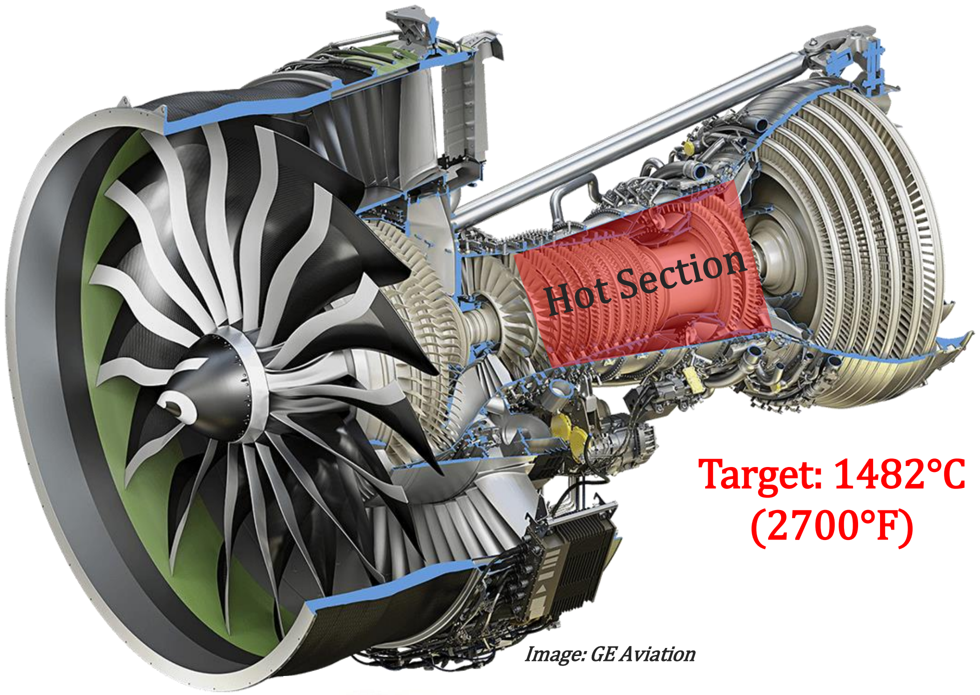
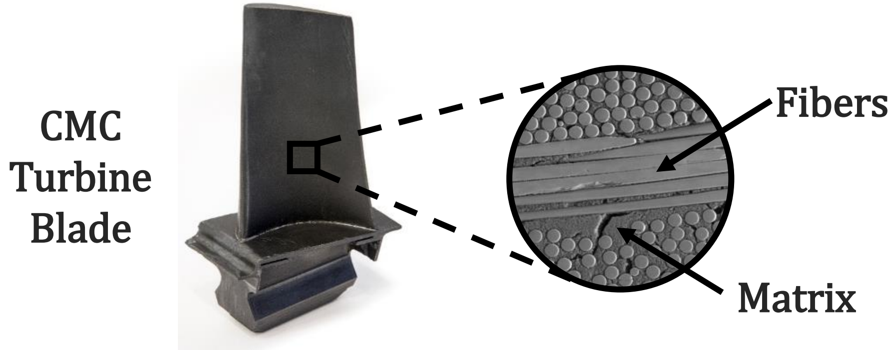


Image: GE Aviation



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Intrinsic Material Selection Criteria

- **Coefficient of thermal expansion (CTE)**
- **Sintering resistance**
- **Low H_2O and O_2 diffusivity/solubility**
- **Phase Stability**
- **Low Modulus**
- **Limited coating interaction**

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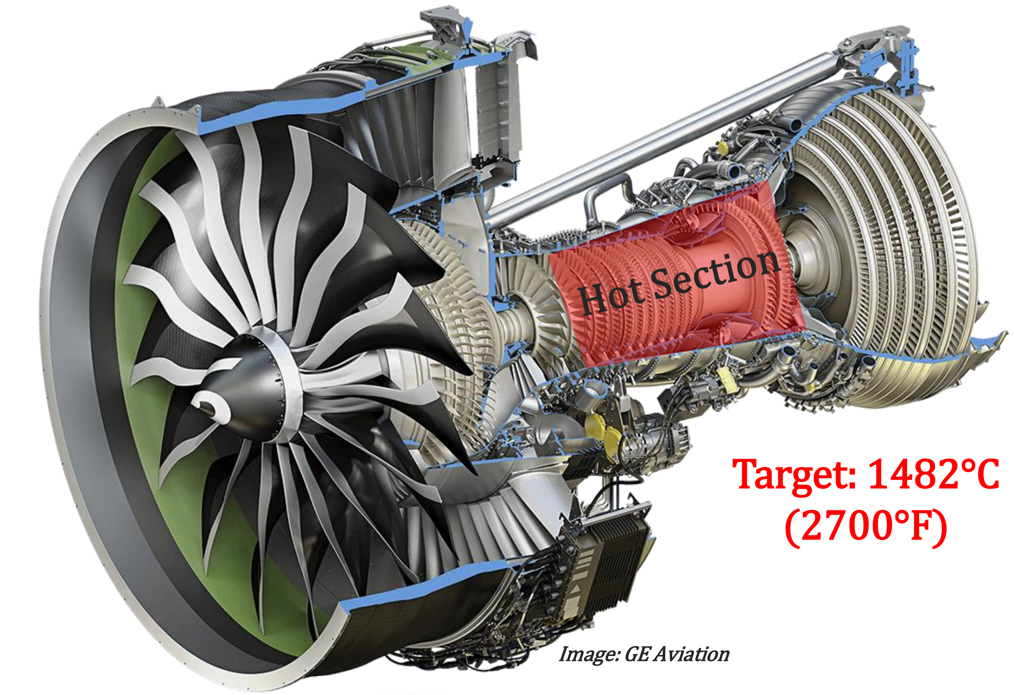

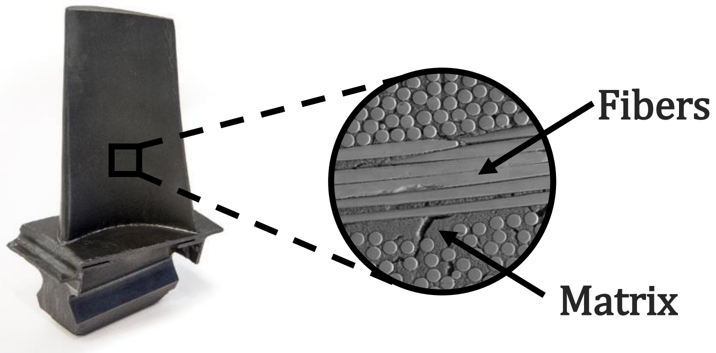

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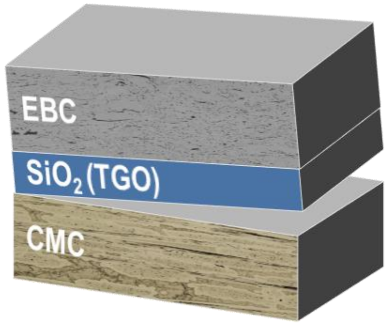
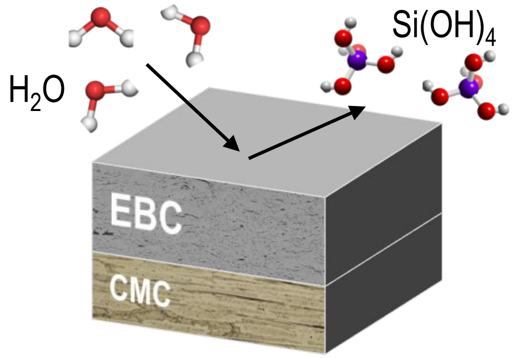
$Yb_2Si_2O_7$

Properties	Ytterbium Disilicate
CTE (x 10^{-6}/K)	4.0
Elastic Modulus (GPa)	168
Melting Temperature (°C)	1850



Steam Oxidation

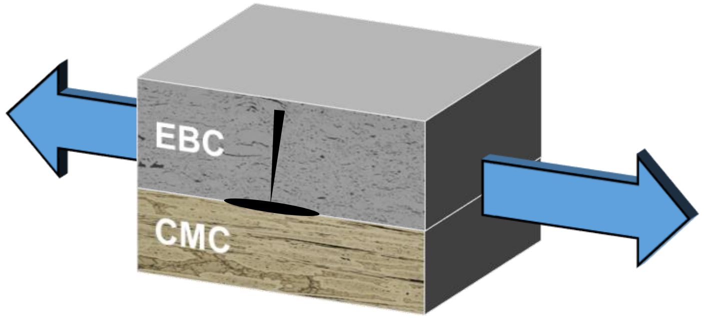


Hydroxide Formation/Recession

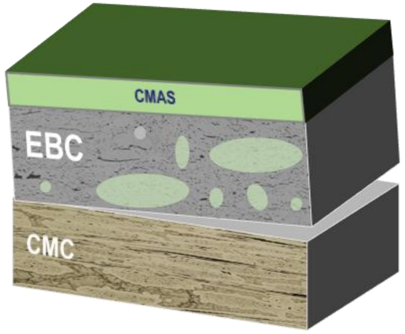
Testing of EBC systems is critical

Individual mechanisms must be well understood before evaluating combinatorial effects

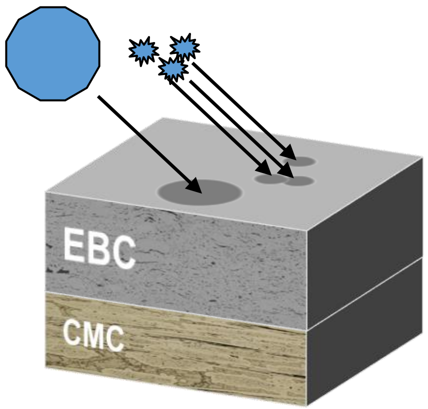
Synergies between extrinsic failure modes determine EBC lifetime and design requirements



Thermomechanical Durability



Molten Silicate Attack and Infiltration



Erosion and FOD

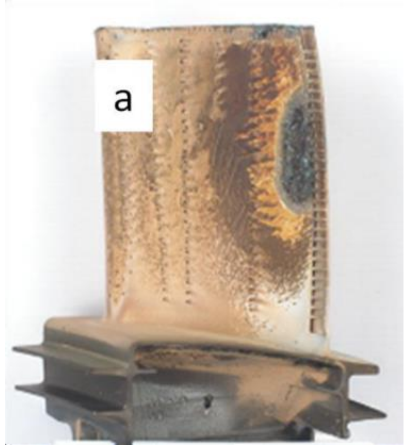
Lee, "Environmental Barrier Coatings for CMCs"; in Ceramic Matrix Composites, (2015)

Introduction and Motivation – EBC Degradation by Particulate Ingestion

CMAS

- Particulates (i.e. sand, volcanic ash) ingested by engine melt into **<u>C</u>alcium-<u>M</u>agnesium-<u>A</u>lumino-<u>S</u>ilicate (CMAS)** deposits above 1200°C
- Molten CMAS degrades EBCs (chemical + mechanical)
  - CMAS infiltration of EBC due to lowered CMAS viscosity at elevated temperatures → CTE mismatch
  - Thermochemical interactions of CMAS with EBC → spallation

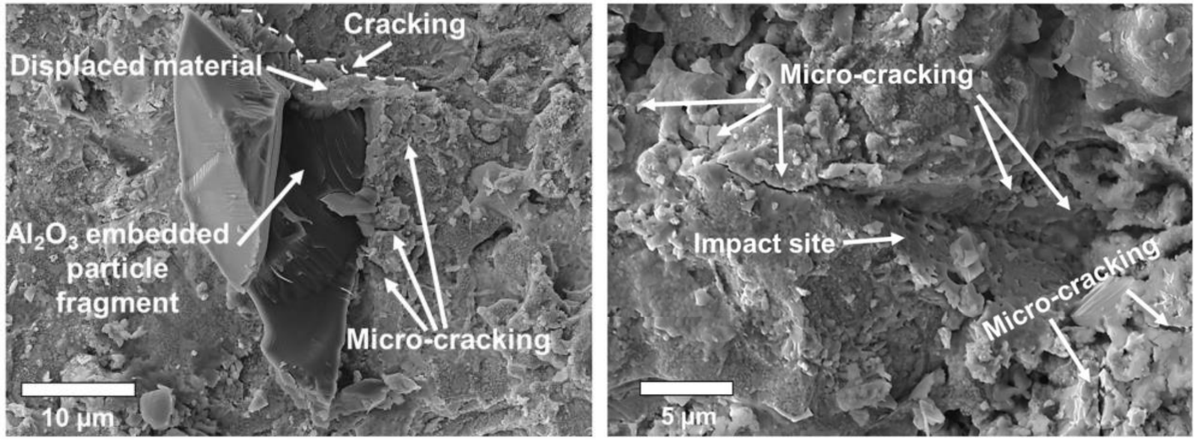
Eyjafjallajökull volcano eruption in Iceland (2010)



Damage on a turbine blade caused by CMAS >1200°C

Solid Particle Erosion

- Particulates (i.e. sand, volcanic ash) ingested by engine can **mechanically erode** EBCs and CMCs at higher temperatures
- Brittle fracture dominated erosion response of EBCs at high temperature
  - Coating microstructure affects durability



Presby et al., *Ceramics International* **47** (2021)

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How is erosion durability affected by microstructural and chemical changes caused by CMAS exposure?

Eyjafjallajökull volcano eruption in Iceland (2010)

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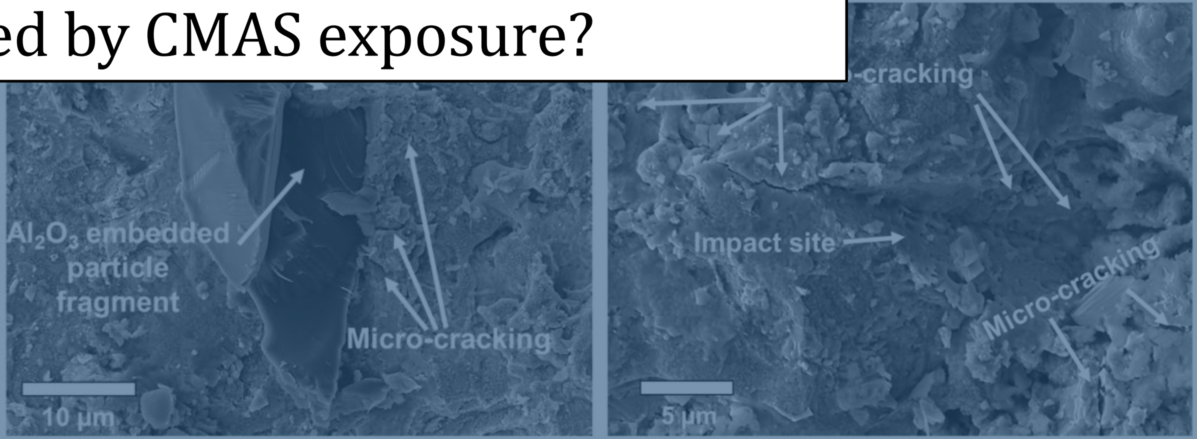
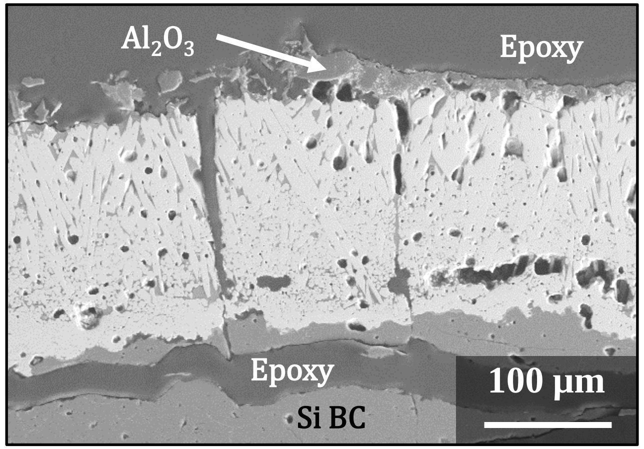


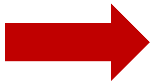
Presby et al., *Ceramics International* **47** (2021)

Previous Work and Outstanding Questions

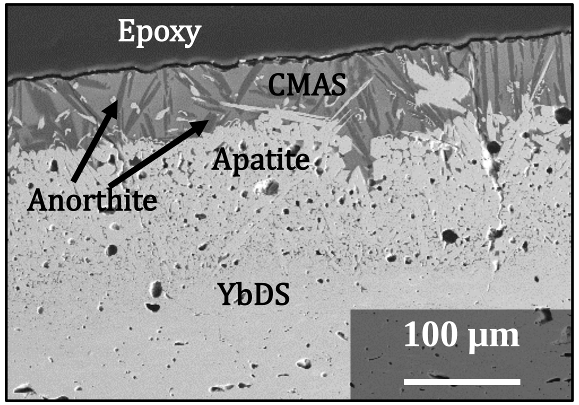
- Damage observed during erosion testing was primarily caused by implications of CMAS reactivity and not particle impacts, making erosion behavior difficult to assess.

Erodent accumulation and catastrophic coating failure





Morphological changes affecting mechanical durability?



Cracking in the bond coat due to CTE mismatch between the coating and residual glass, as well as catastrophic coating failure due to thermal shock in the burner rig

Differences in the thermal and mechanical properties (thermal expansion, fracture toughness, elastic modulus, hardness) of reaction products will affect durability

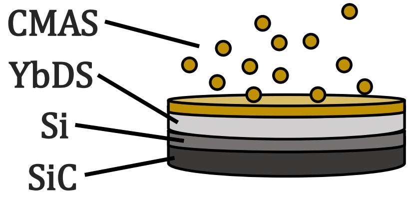
Stokes et al., *Proceedings of ASME Turbo Expo 2023 Turbomachinery Technical Conference and Exposition* (2023)

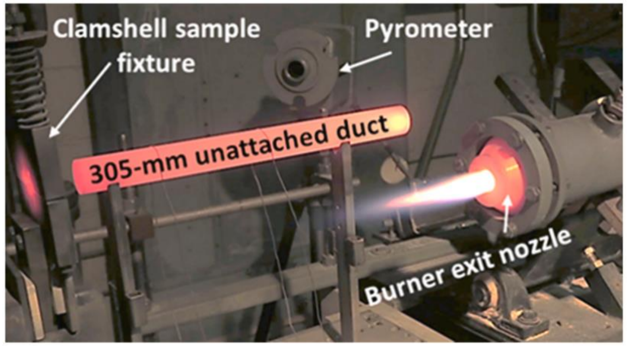
Experimental Procedures

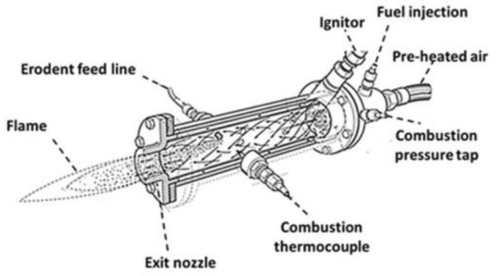
- Two air plasma sprayed $Yb_2Si_2O_7$ (YbDS) coatings: baseline $Yb_2Si_2O_7$ and modified (M2Y) $Yb_2Si_2O_7$
  - The modified coating has YAG, mullite, added to improve oxidation performance; Lee, *J. Am. Cer* **102** (2019)
  - ~250 µm topcoat with ~125 µm Si bondcoat on SiC SA Hexoloy
- ~2 and ~4 mg/cm^2 loadings
  - $30.67CaO–8.25MgO–12.81AlO_{1.5}–48.27SiO_2$ (mol.%)
  - Krämer et al. *J. Am. Cer.* **89** (2006)
  - Applied by air spray (Harder et al. *J. Eur. Cer.* ***44*** *2024*)
- All samples furnace heat treated at 1316°C, 4 hours;
- Reaction products identified using SEM/EDS
- Erosion testing carried out in NASA's Erosion Burner Rig Facility at 1316°C
- ~60 µm Al_2O_3 erodent; 30°, 60° and 90° impingement angle

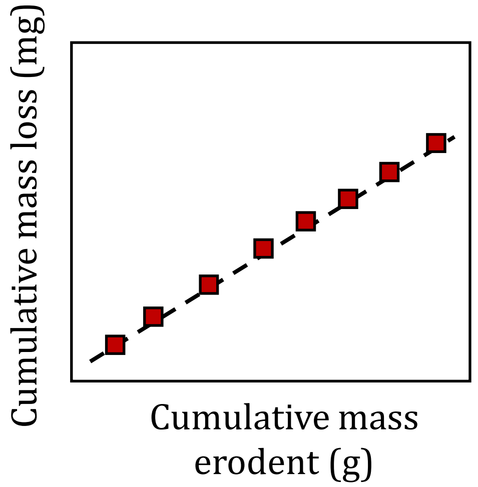
Air spray

~2 and ~4 mg/cm^2







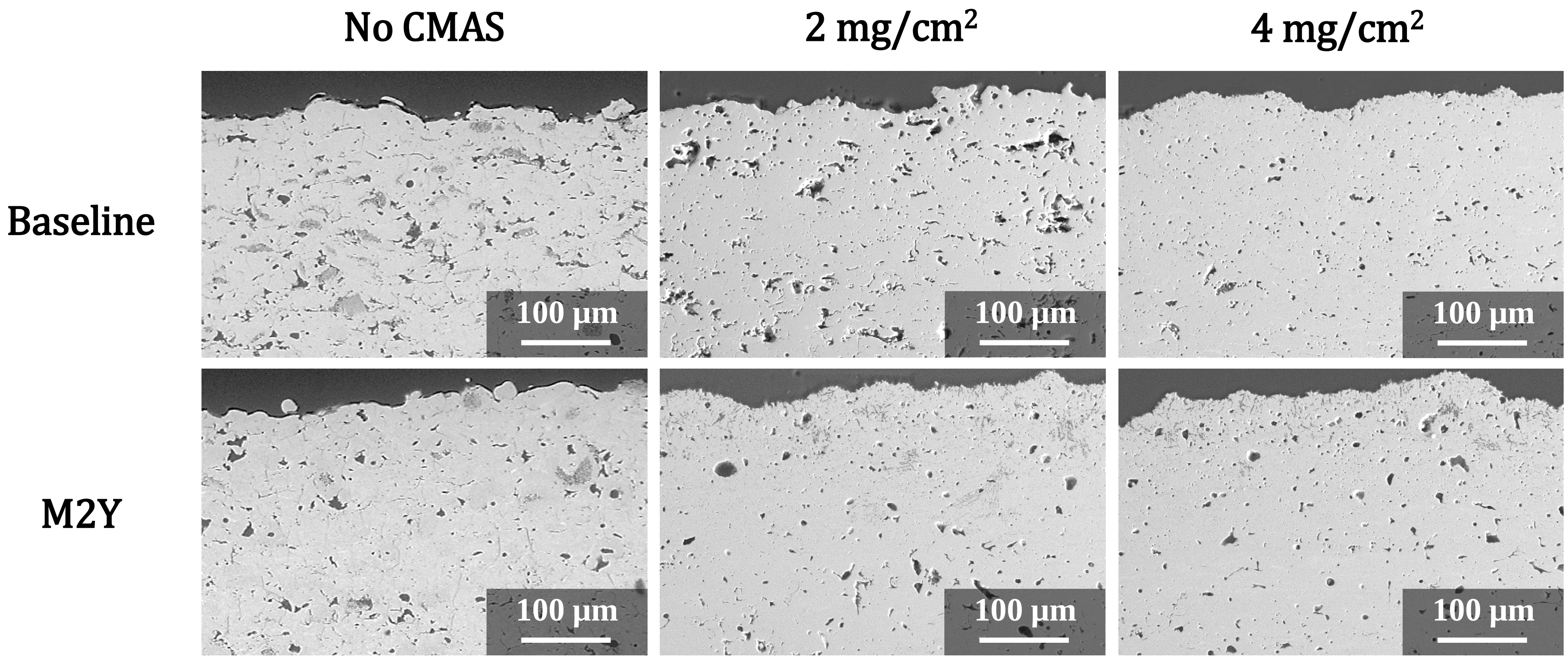


D.S. Fox et al., NASA/TM-2011216986 (2011)

Results – Starting Morphology

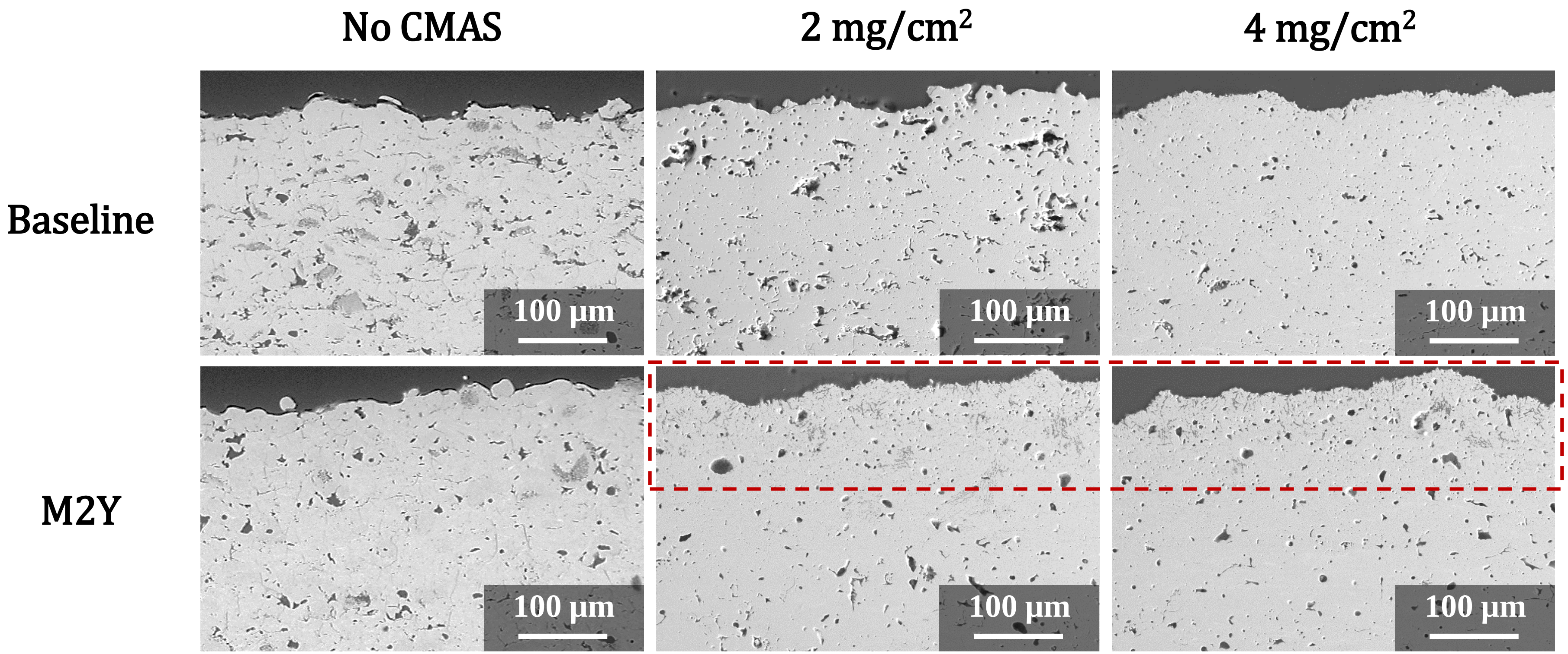
- Lamellar cracks and porosity characteristic of APS coatings were apparent in the as-deposited coatings, although many of those morphological features were no longer present after the coatings were exposed to CMAS.

- There was no residual CMAS remaining on the surface of the coatings.



Results – Starting Morphology

- Distinct CMAS reaction layer at the surface
- Higher magnification shows formation of $Ca_2Yb(SiO_4)_6O_2$ apatite (needle-like precipitates)



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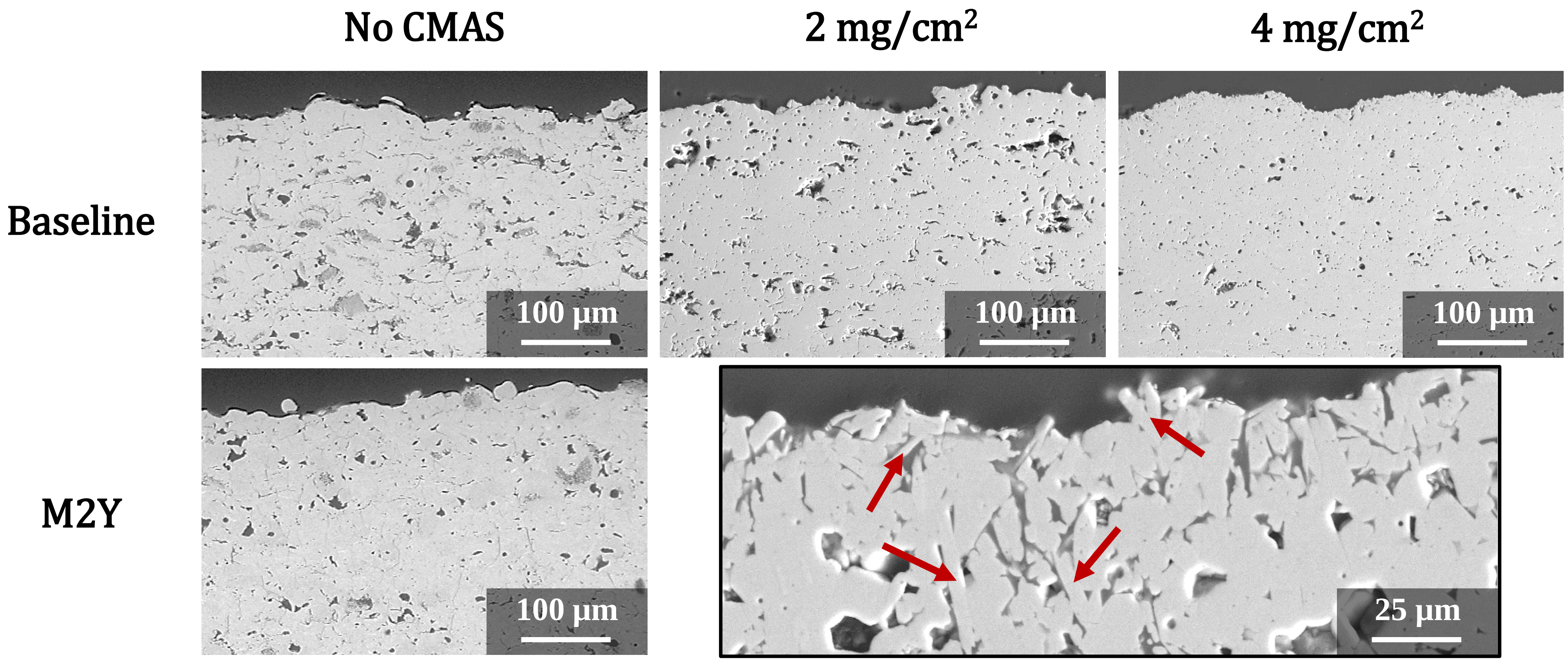

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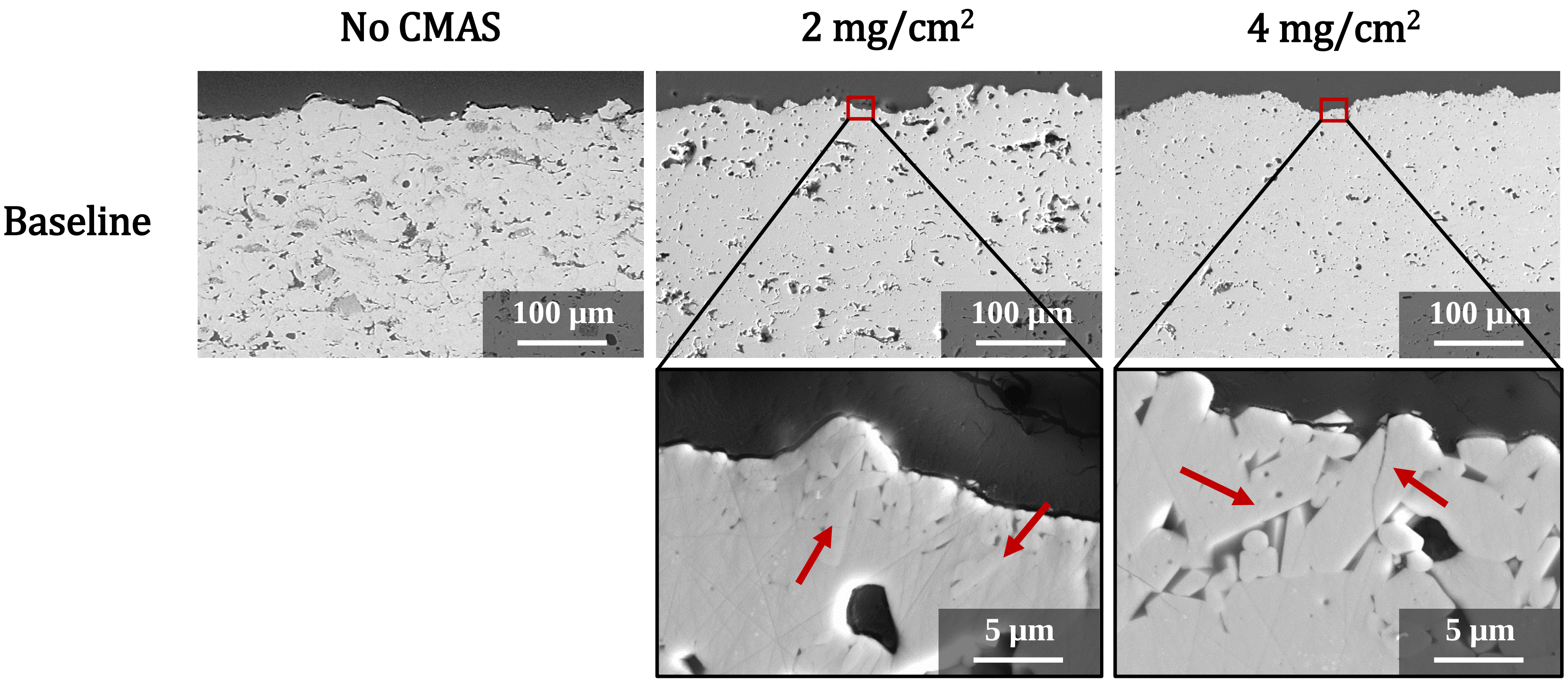
Results – Starting Morphology

- Baseline sample also exhibit apatite formation in interaction region at surface much thinner than M2Y



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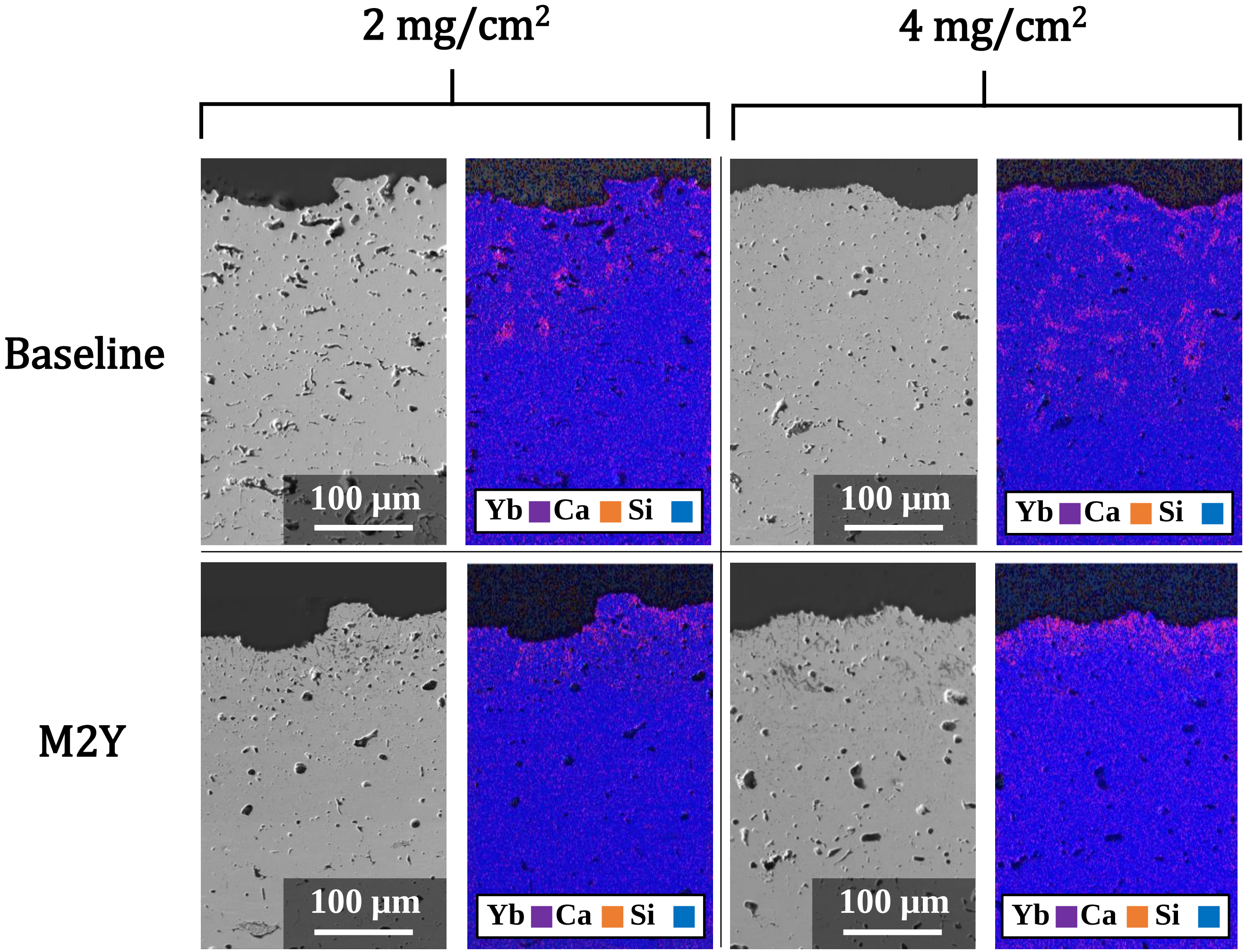
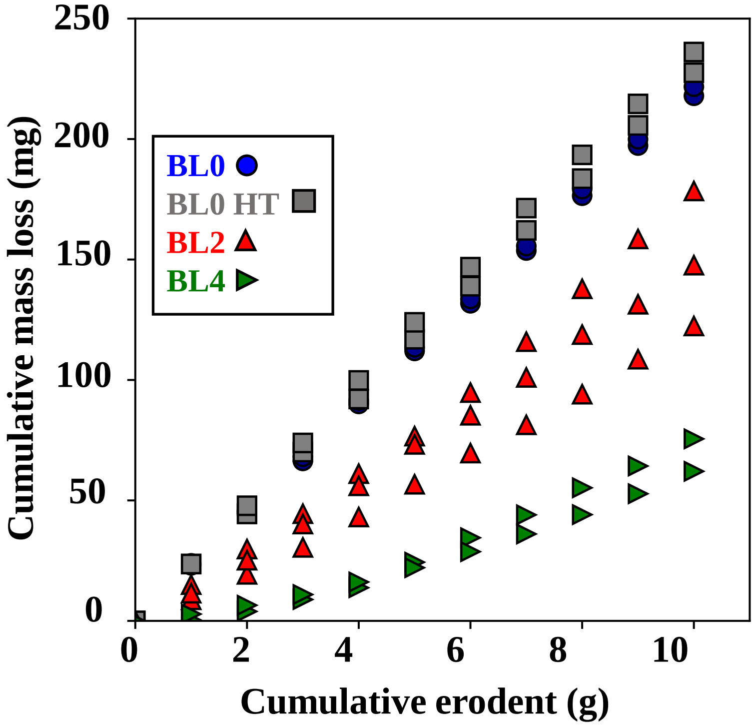
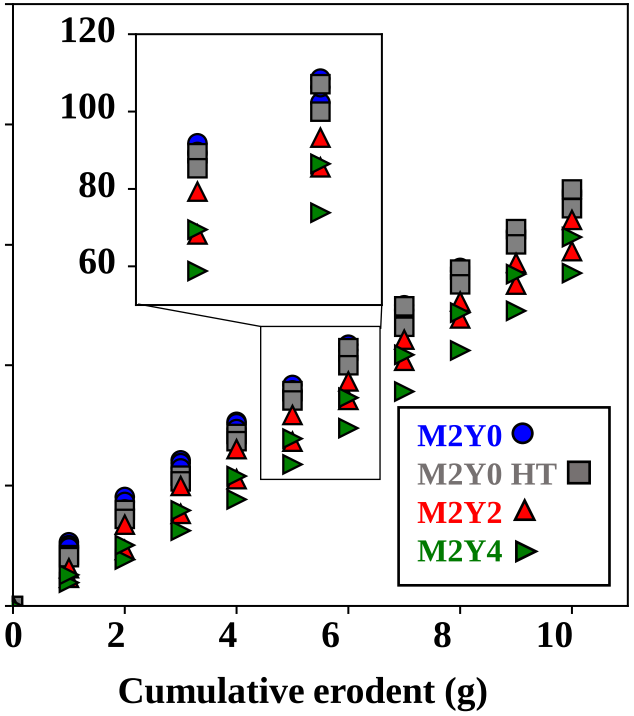


- Thin region at the surface of the coating rich in both Ca and Yb, in addition to precipitates scattered throughout the coating with a similar composition, consistent with the formation of apatite.
  - Precipitates found ~140 µm deep in 2 mg/cm$_2$ sample, ~230 µm deep in 4 mg/cm$_2$ sample).
  - Precipitates accounted for ~3% areal fraction of the 2 mg/cm^2 sample and ~12% areal fraction of the 4 mg/cm^2 sample.

- Apatite precipitates were much smaller at concentrated at surface of the M2Y coatings

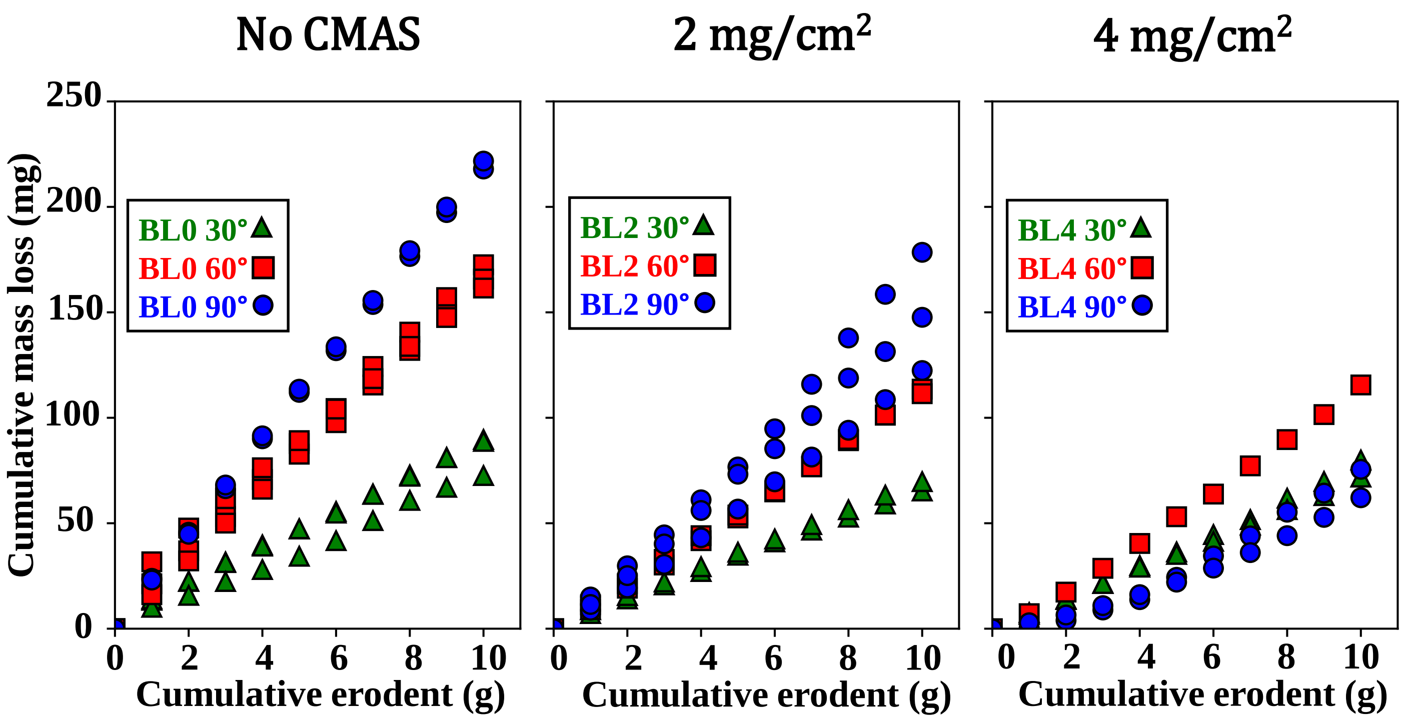
Erosion Results – 90° Impingement Angle





- For all coatings, there was decrease in cumulative mass loss with increase CMAS loading, although the differences in mass loss were wider for the baseline coatings than the M2Y coatings.
- In the baseline 2 mg/cm^2 and 4 mg/cm^2 samples, the plots exhibited some non-linear behavior, having curvature at ~5-6 g cumulative erodent.
- Heat treatment of both the baseline and M2Y coatings at 1316°C, 4 hours with no CMAS resulted in erosion behavior equal to the as deposited coatings

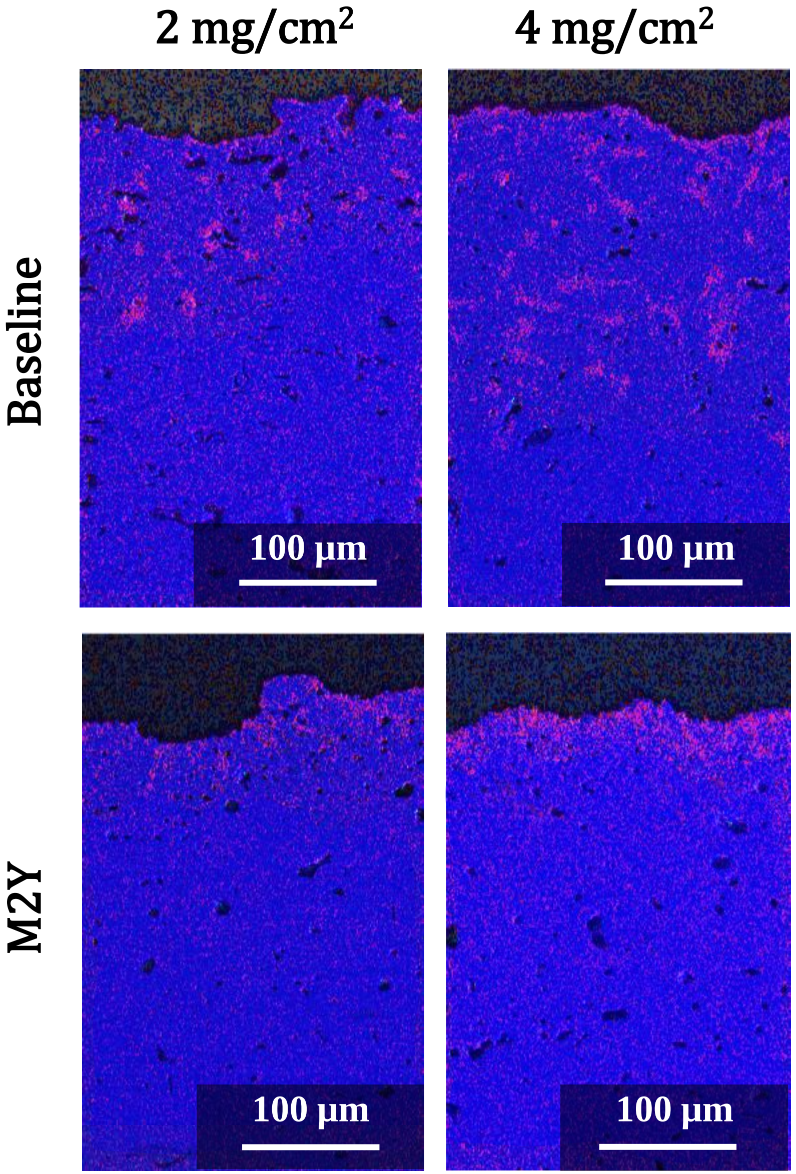
Erosion Results – Effect of Impingement Angle on Baseline Samples



- In both the baseline coating not exposed to and the 2 mg/cm^2 sample, the cumulative mass loss increased with increasing impingement angle.
- In the case of the 4 mg/cm^2 sample, the greatest cumulative mass loss was observed for the sample tested at 60°.

Erosion Results – Apatite Formation Improved Erosion Durability

- CMAS interactions with the coatings resulted in improvement in solid particle erosion durability
- Effect was greater in the case of the baseline YbDS coatings over the M2Y coatings.
  - thermochemical reaction with CMAS resulted in formation of $Ca_2Yb_8(SiO_4)_6O_2$ apatite
  - Apatite formed as small precipitates within a reaction layer at the surface of the M2Y coatings.
  - Larger apatite inclusions formed throughout the baseline coatings



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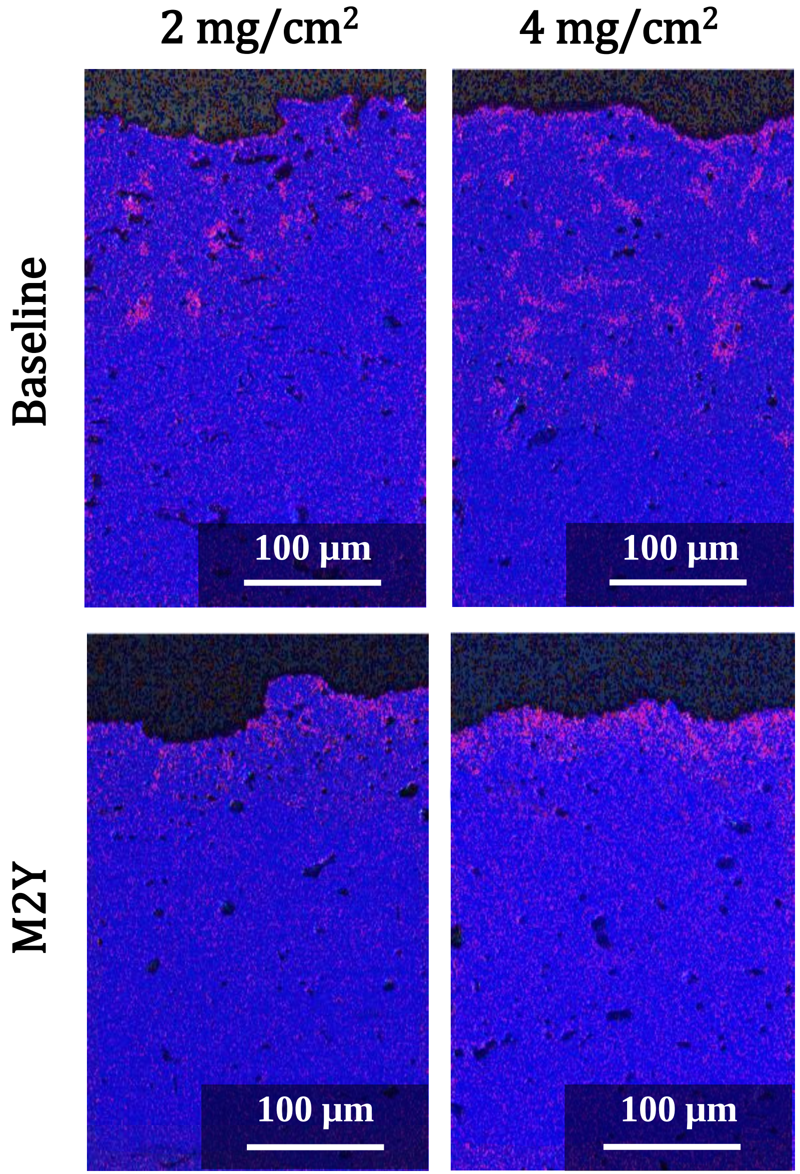
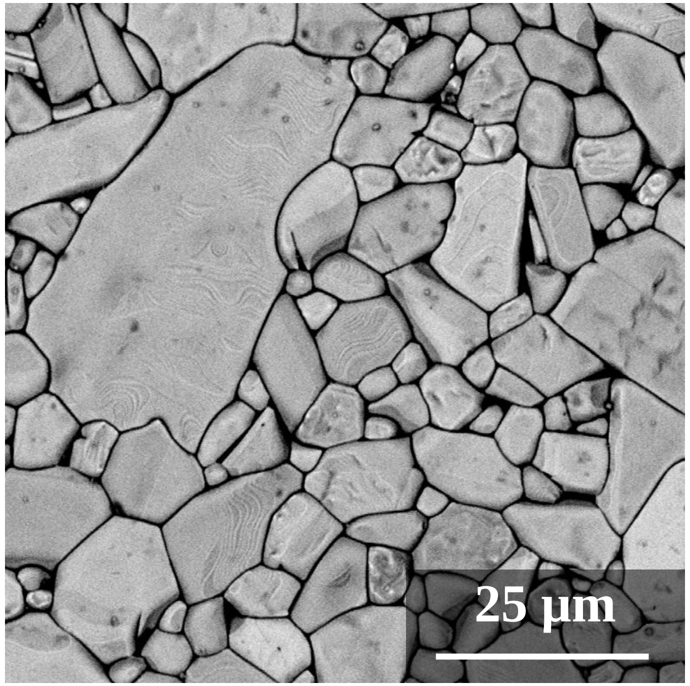
- Why has apatite formation improved erosion durability?
  - Does apatite have mechanical properties that give the phase better durability against particulate erosion?



Mechanical Properties of $Yb_2Si_2O_7$ and $Ca_2Yb_8(SiO_4)_6O_2$

- Bulk pellets were synthesized of to determine the mechanical properties of both materials.

$Yb_2Si_2O_7$



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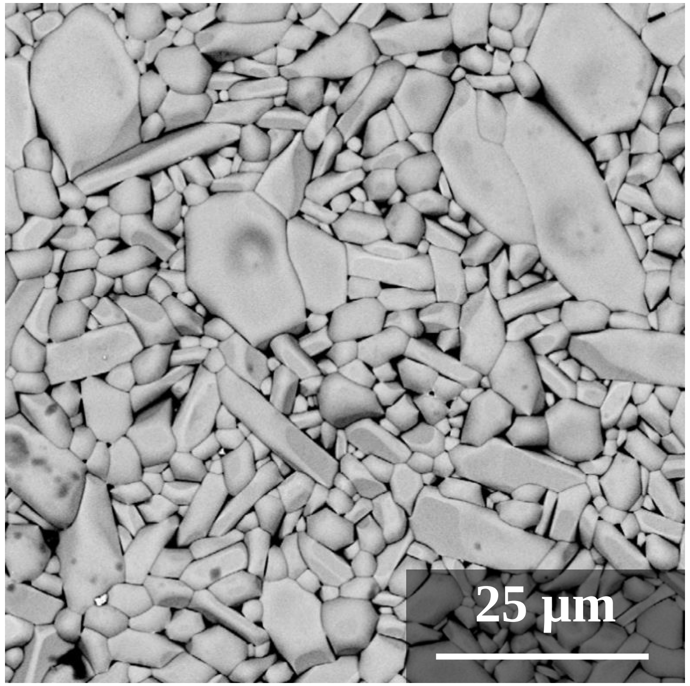
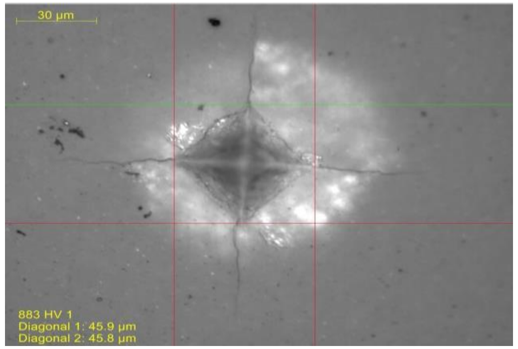


- Relationship between mechanical properties and erosion response

$$V \propto U_k^{7/6} E_t^{5/4} K_{IC,t}^{-1} H_t^{-17/12}$$

- Vickers indentation for hardness and fracture toughness indents



- Impulse excitation to measure elastic modulus

Marshall et al. *J. Am. Ceram. Soc.* **65** (1982)
ASTM Standard E384-22
Anstis et al. *J. Am. Ceram. Soc.* **64** (1981)

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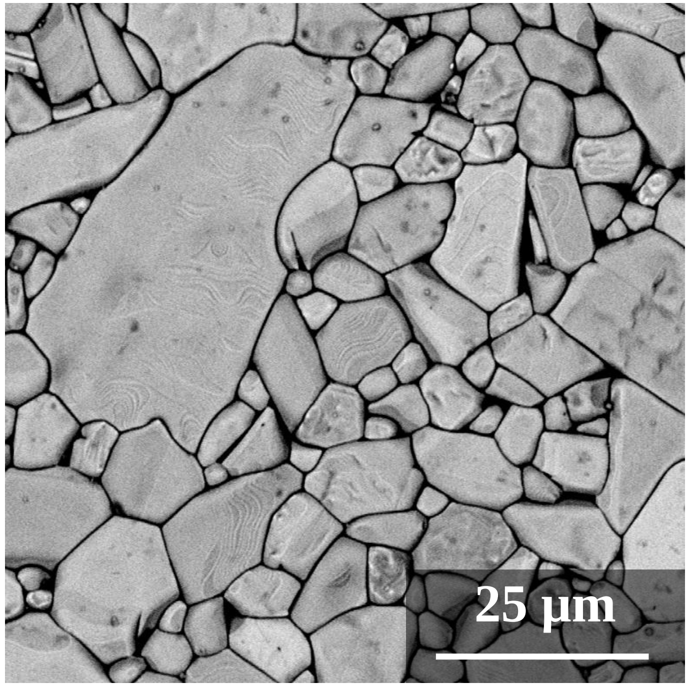

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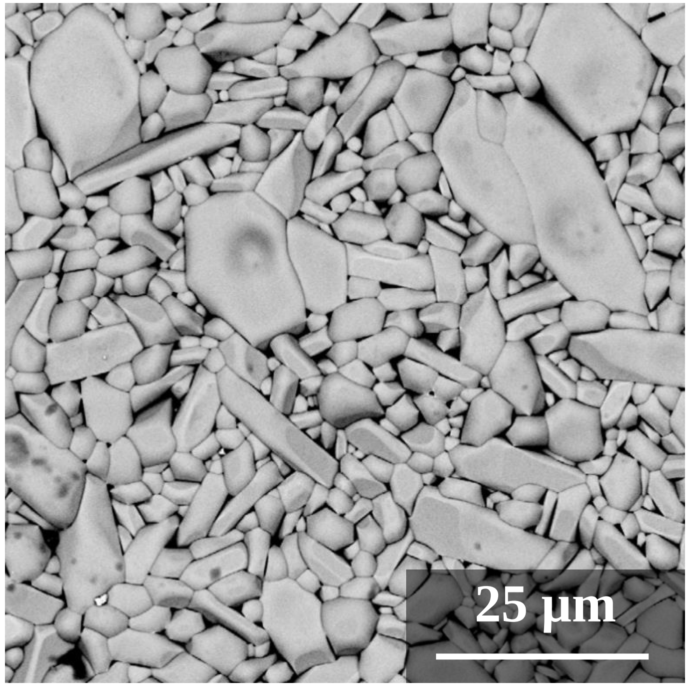

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	E (GPa)	H (GPa)	K_{IC}(MPa·m$^{1/2}$)
$Yb_2Si_2O_7$	152±2	8.83±0.27	1.32±0.05
$Ca_2Yb_8(SiO_4)_6O_2$	138±1	7.46±0.32	0.99±0.11

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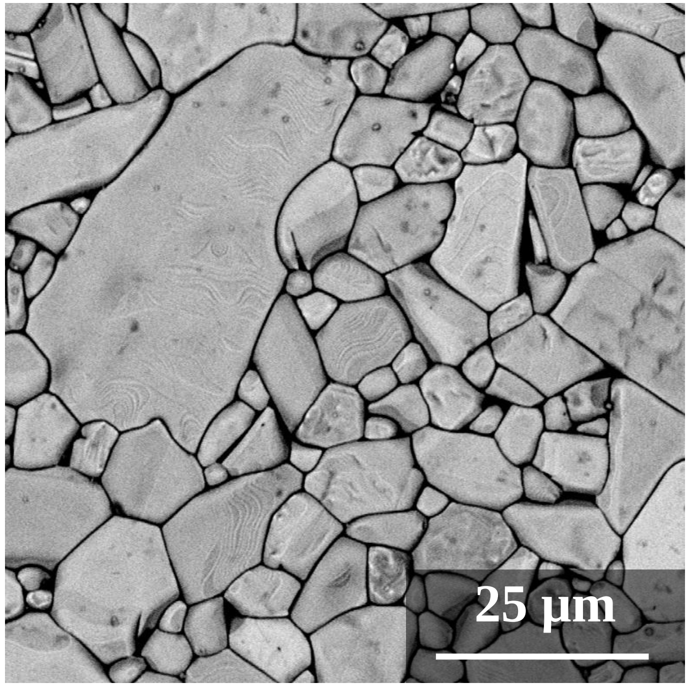

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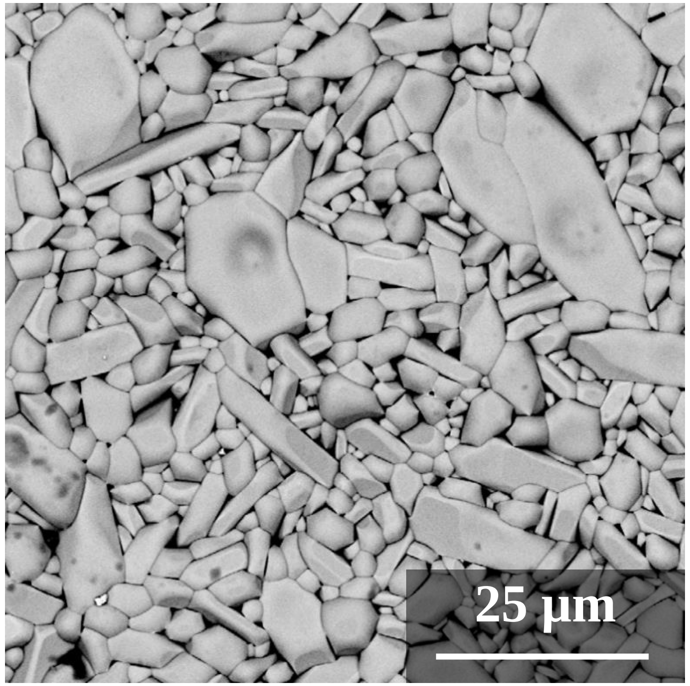

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- Theoretical volume loss
  - $Yb_2Si_2O_7$ 3.00×10^{-13} m^3
  - $Ca_2Yb_8(SiO_4)_6O_2$ 4.51×10^{-13} m^3

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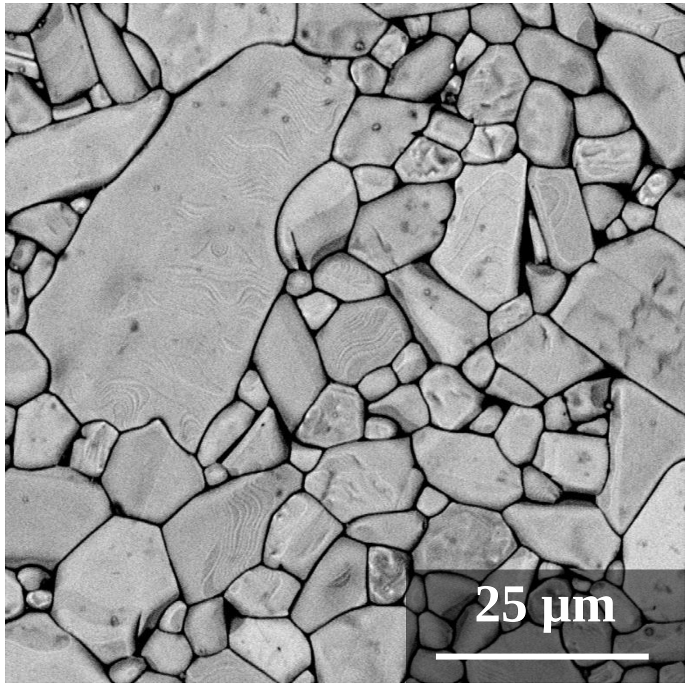
Mechanical Properties of Composites

- What unique properties of composites improve mechanical strength?

$Yb_2Si_2O_7$



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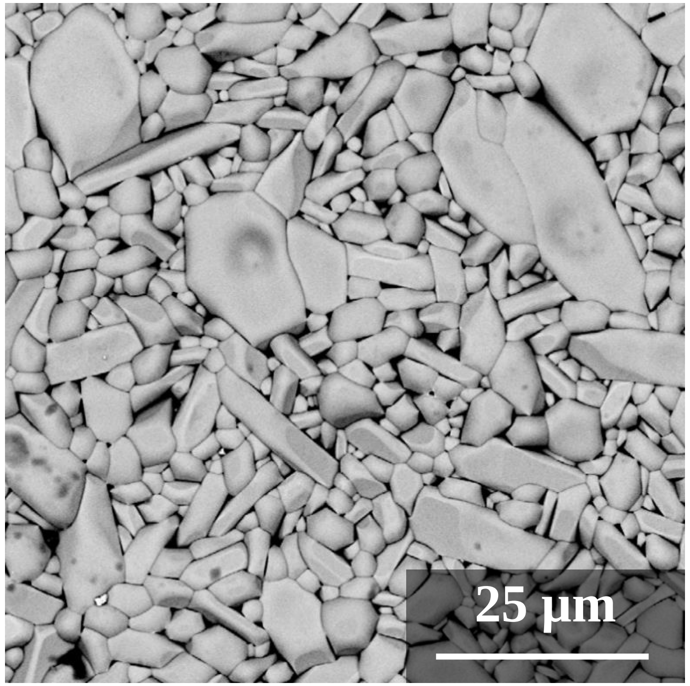
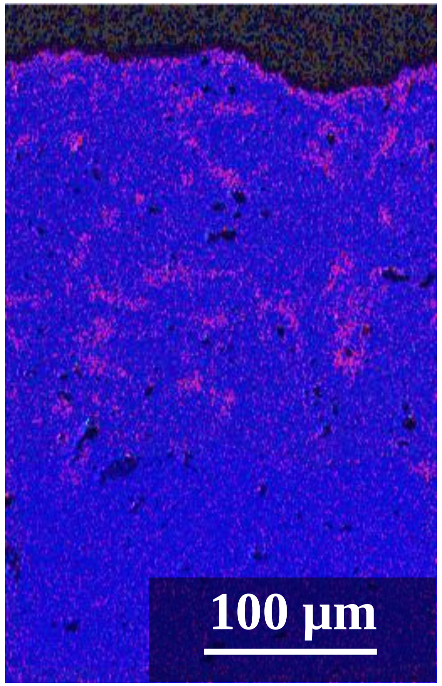
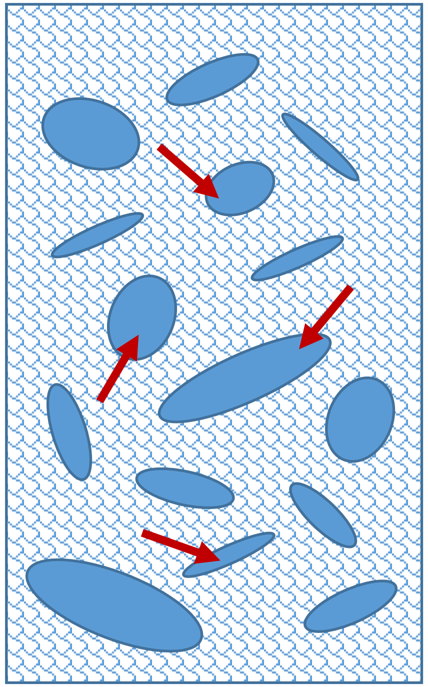




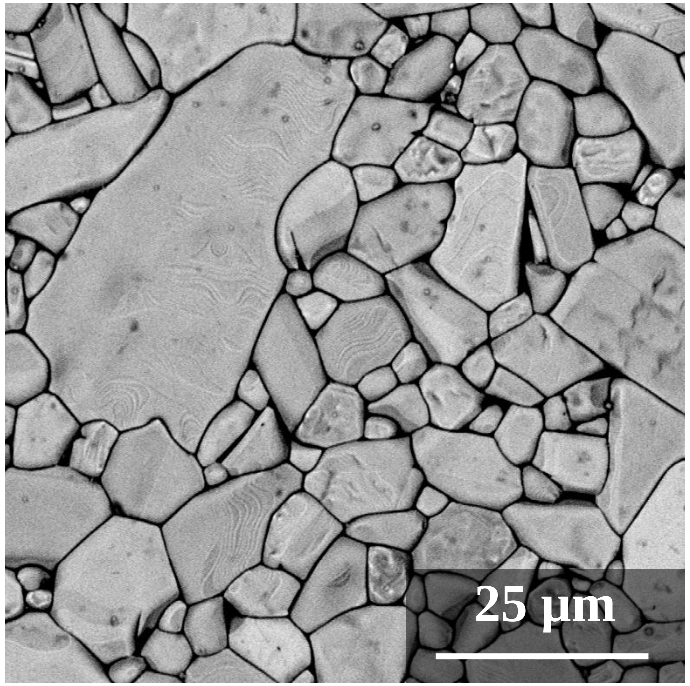
Baseline, 4 mg/cm^2



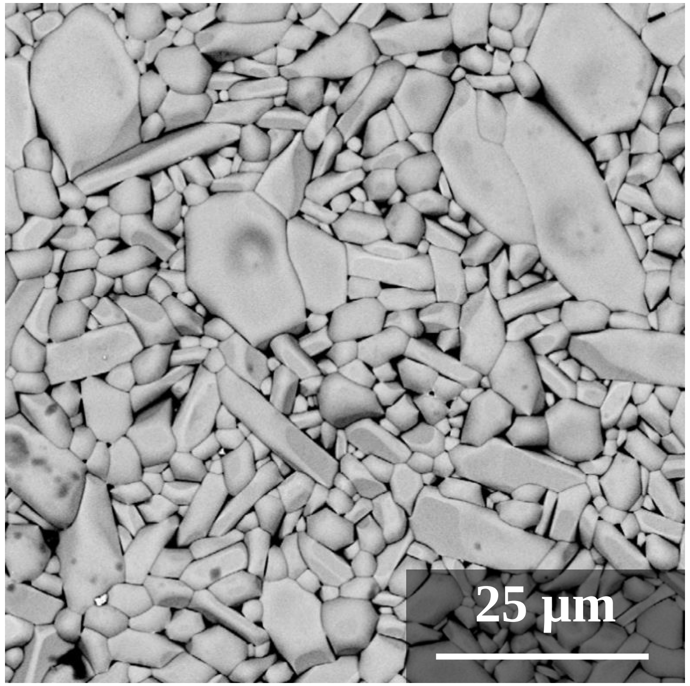
Mechanical Properties of Composites

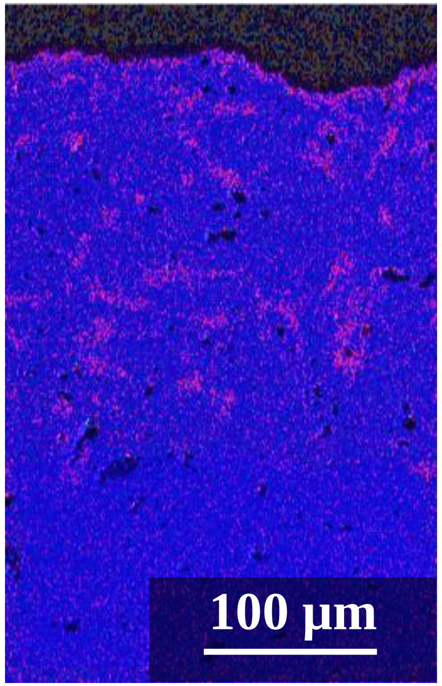
- Thermal expansion mismatch strengthening?

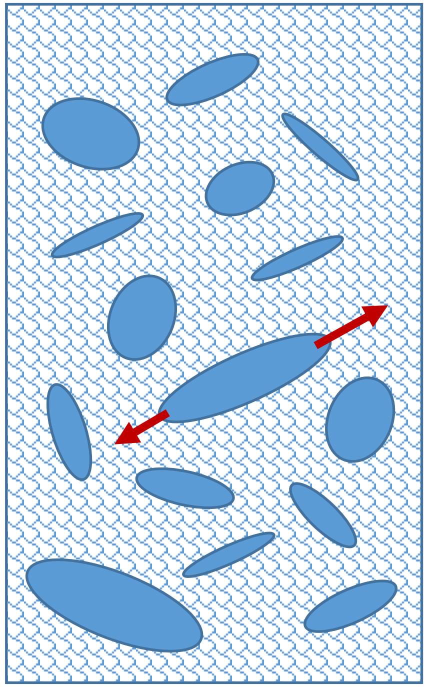
$Yb_2Si_2O_7$



$Ca_2Yb_8(SiO_4)_6O_2$







Baseline, 4 mg/cm^2

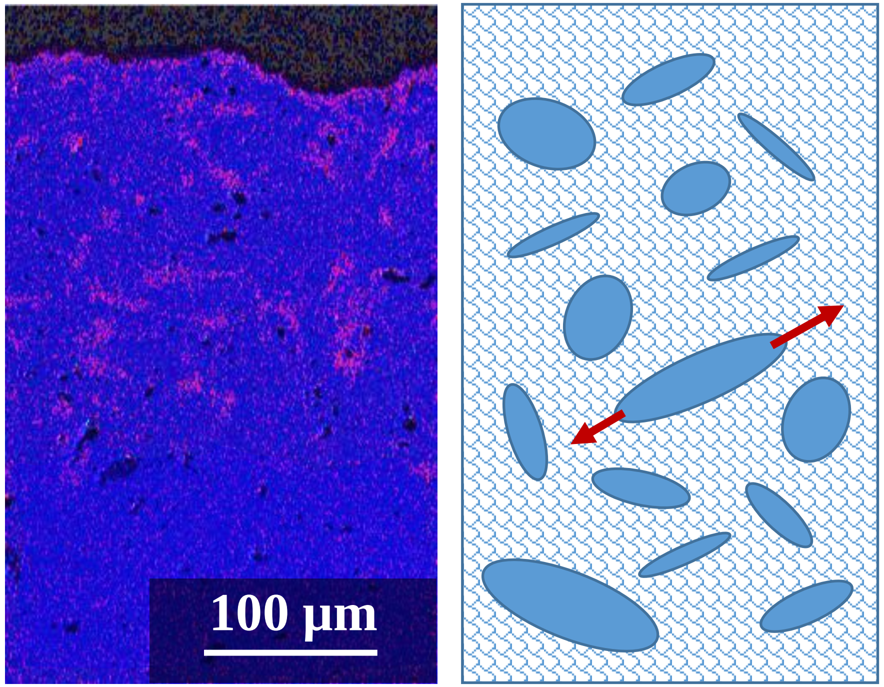
- $Yb_2Si_2O_7$ ~4×10^{-6}/K
- $Ca_2Yb_8(SiO_4)_6O_2$ ~8.5×10^{-6}/K

J.L. Stokes, NASA/TM-20210009690 (2021)

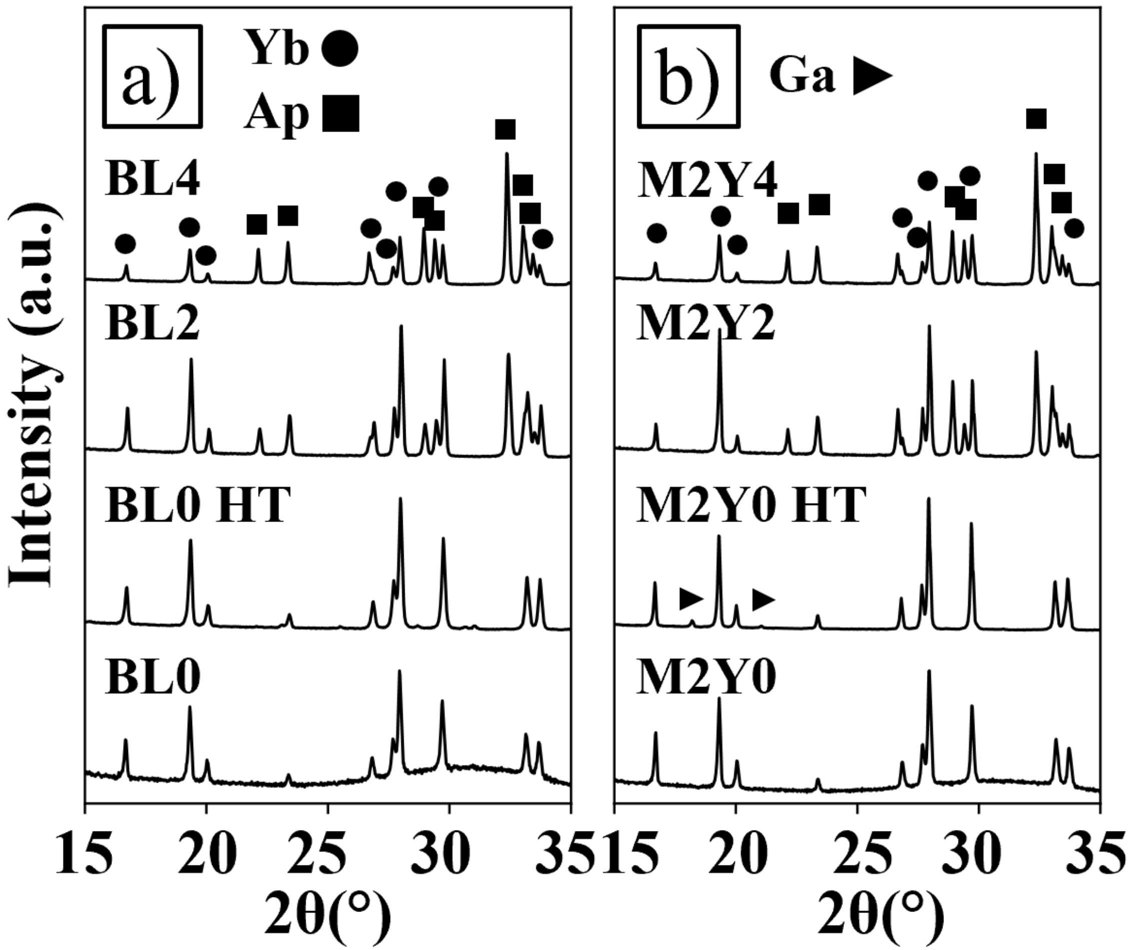
Summary and Future Work

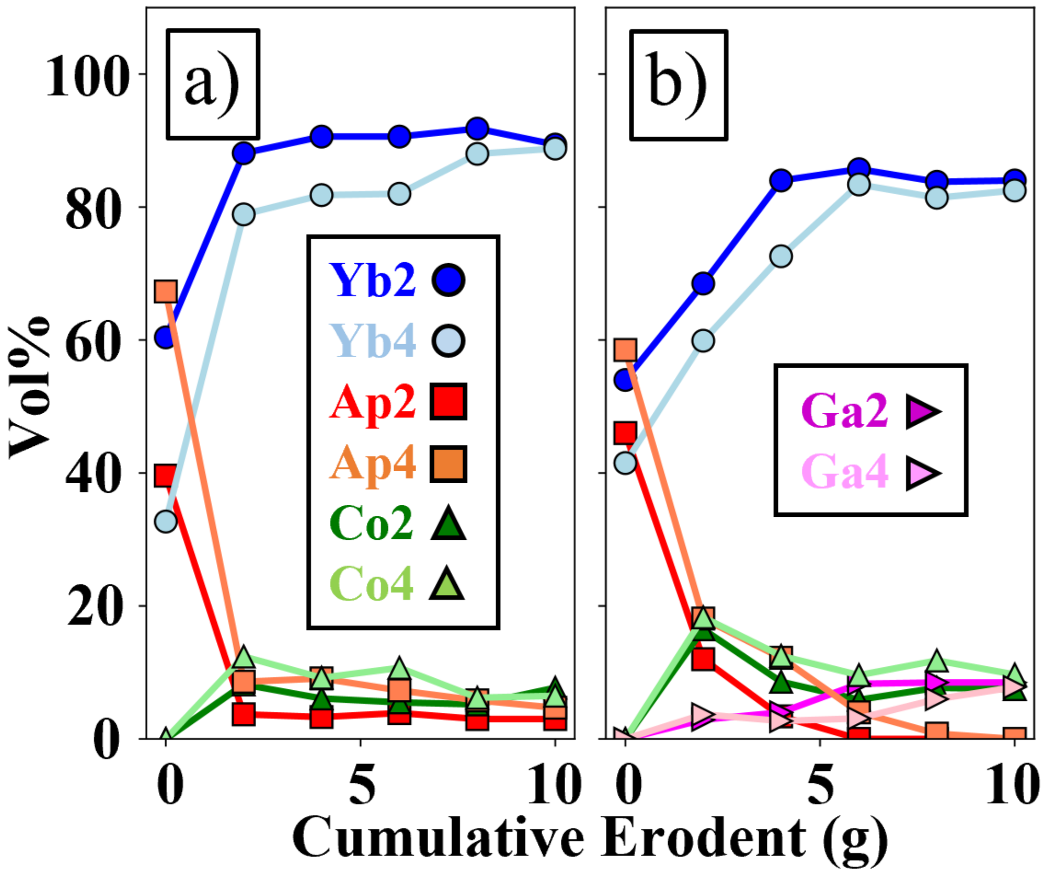
- Particulate/debris damage caused by ingestion of CMAS hinders the use of environmental barrier coatings (EBCs) to protect CMCs.
- This study assessed the effects of extrinsic phase formation and microstructural changes due to CMAS interactions on the erosion durability of $Yb_2Si_2O_7$-based EBCs
  - Apatite morphology differed based on coating chemistry
  - Apatite formation improved erosion durability
  - Could the improvement be caused by thermal expansion mismatch strengthening?
- Testing bulk pellets of $Yb_2Si_2O_7$, $Ca_2Yb_8(SiO_4)_6O_2$, and a composite disilicate-apatite pellet in NASA Glenn's Erosion Burner Rig Facility to determine mechanism of strengthening in the coating samples

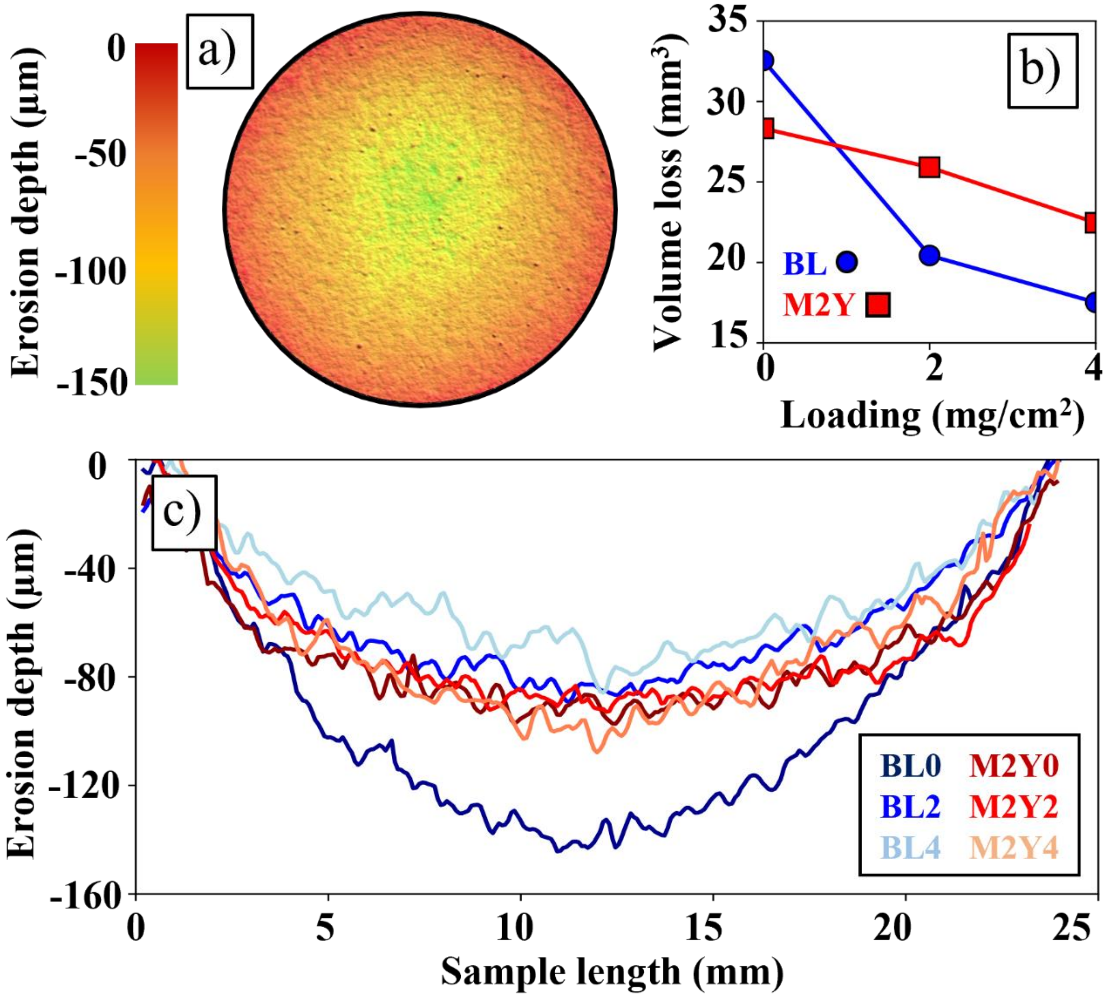
Baseline, 4 mg/cm^2

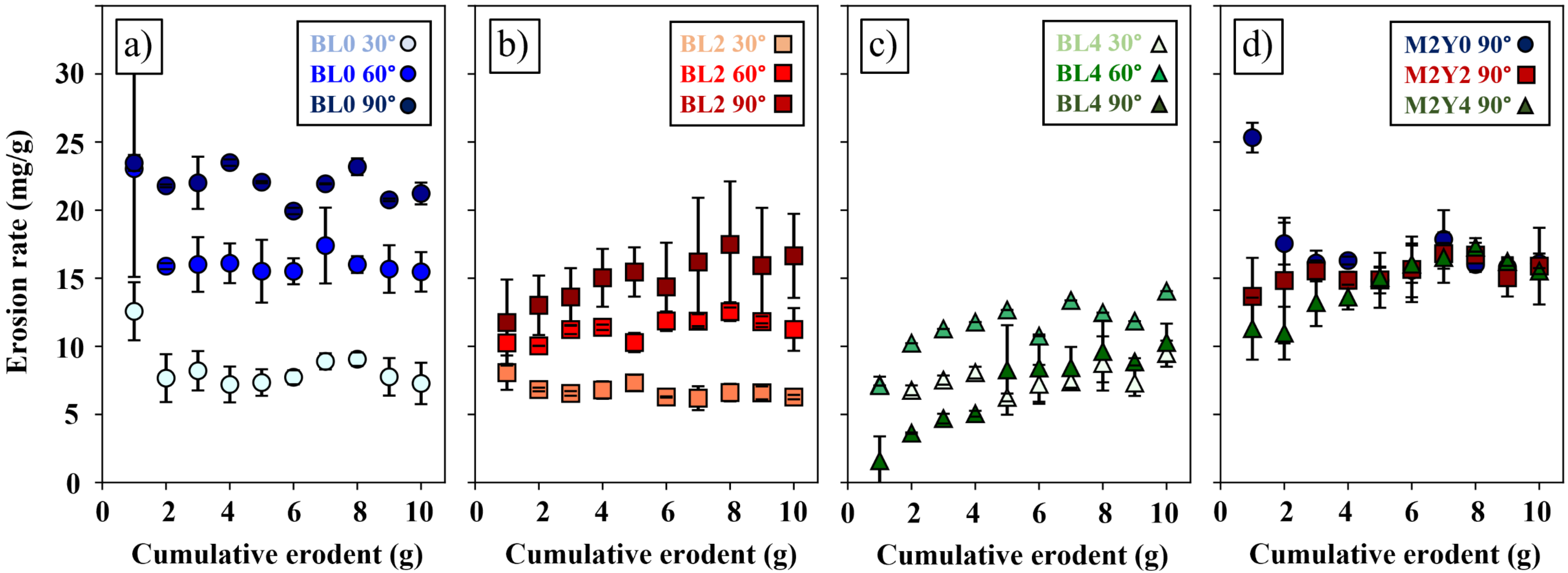


Thank You!


a)
Yb ●
Ap ■
BL4
BL2
BL0 HT
BL0
Intensity (a.u.)
15 20 25 30 35
2θ(°)
b)
Ga ▶
M2Y4
M2Y2
M2Y0 HT
M2Y0
15 20 25 30 35
2θ(°)


a)
b)
100
80
60
40
20
0
Vol%
0
5
10
Cumulative Erodent (g)
Yb2
Yb4
Ap2
Ap4
Co2
Co4
Ga2
Ga4


a)
Erosion depth (µm)
0
-50
-100
-150
b)
Volume loss (mm³)
Loading (mg/cm²)
BL
M2Y
c)
Erosion depth (µm)
Sample length (mm)
BL0
BL2
BL4
M2Y0
M2Y2
M2Y4


a)
BL0 30°
BL0 60°
BL0 90°
b)
BL2 30°
BL2 60°
BL2 90°
c)
BL4 30°
BL4 60°
BL4 90°
d)
M2Y0 90°
M2Y2 90°
M2Y4 90°
Erosion rate (mg/g)
Cumulative erodent (g)
0
5
10
15
20
25
30
0
2
4
6
8
10